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Short communication

Different relative rates for photo-rearrangements of (*E*)- and (*Z*)-β-nitrostyrene derivatives to oximinoketones

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Abstract

New details on photochemistry of β-nitrostyrene derivatives are found, using a 500 MHz NMR spectrometer, instead of the formerly employed UV spectroscopy, where simply photo-rearrangement of (*E*)-β-methyl-β-nitrostyrene, $\mathbf{1}_E$, to 1-phenyl-1,2-propanedione-1-oxime, **2**, as well as *cis–trans* isomerization of (*E*)-β-nitrostyrene, $\mathbf{3}_E$, to (*Z*)-β-nitrostyrene, $\mathbf{3}_Z$, were reported. Here, using similar conditions such as light intensity, relative rates are found for *cis–trans* isomerization of $\mathbf{1}_E$ to (*Z*)-β-methyl-β-nitrostyrene, $\mathbf{1}_Z$; as well as novel photo-rearrangements of: $\mathbf{1}_Z$ to **2**; $\mathbf{3}_Z$ to 1-phenyl-1,2-ethandione-1-oxime, **4**; and $\mathbf{3}_E$ to **4**. ^{1}H NMR preliminary kinetic analysis show isomerization of $\mathbf{1}_E$ to $\mathbf{1}_Z$ occurring at a relative rate of $k_{\text{i-Me}} = 0.083\ \text{s}^{-1}$. Both $\mathbf{1}_E$ and $\mathbf{1}_Z$ go through a nitro-nitrite photo-rearrangement to **2** with relative $k_{\text{r-Me}} = 0.011\ \text{s}^{-1}$ and $k'_{\text{r-Me}} = 0.070\ \text{s}^{-1}$, respectively. Under the same conditions, photo-isomerization of $\mathbf{3}_E$ to $\mathbf{3}_Z$ takes place at relative $k_{\text{i-H}} = 0.011\ \text{s}^{-1}$. The rates of photo-rearrangements of $\mathbf{3}_E$ as well as $\mathbf{3}_Z$ to **4** are measured with relative $k_{\text{r}} = 0.025\ \text{s}^{-1}$ and $k'_{\text{r}} = 0.010\ \text{s}^{-1}$, respectively.

Keywords: β-Nitrostyrene; β-Methyl-β-nitrostyrene; *Cis–trans* isomerization; Nitro-nitrite rearrangement; NMR; Rates of reaction; Photo-rearrangement; Nitroolefins

1. Introduction

β-Nitrostyrenes are important intermediates in organic syntheses [1]. They are used as starting materials for many classes of compounds [2–6]. Their photolysis has received considerable attention [7–32]. Solar irradiation of (*E*)-β-methyl-β-nitrostyrene, $\mathbf{1}_E$, results in its geometrical isomer: (*Z*)-β-methyl-β-nitrostyrene, $\mathbf{1}_Z$ (Scheme 1, path a) [23].

Photolysis of $\mathbf{1}_E$ gives 1-phenyl-1,2-propanedione-1-oxime, **2**, in a quantitative yield (Scheme 1, path b) [17]. Substituent effects on photo-rearrangement of $\mathbf{1}_E$ to **2** are reported by us and others [18,24–30]. We found a rather small Hammett ρ of 0.7 which reflects the small sensitivity of such nitro-nitrite photo-rearrangements to the electronic effects of substituents [18].

Up to date, no report on intermediacy of $\mathbf{1}_Z$ in photo-conversion of $\mathbf{1}_E$ to **2** has appeared. Also, photo-rearrangements of neither $\mathbf{1}_Z$ to **2**, nor $\mathbf{3}_E$ to **4** are reported. As a follow up to our β-nitrostyrene mechanistic studies [18–22], here, new insights including relative preliminary kinetic rates for the involvement of the later compounds in the photochemistry of β-nitrostyrene derivatives are reported, using similar conditions such as light intensity (Scheme 1).

2. Experimental

The UV irradiation source employed is a Hanovia type 400 W low-pressure mercury arc lamp. Ultraviolet spectra are recorded on a Shimadzu model UV-200. IR spectra are determined on a Shimadzu model IR-460. NMR spectra are performed on a 500 MHz NMR Bruker and a Jeol FT-NMR, TNM-EX-90A. GC/MS spectra are recorded on Perkin-Elmer 8420 capillary gas chromatograph with ITD Finnigan-Mat Perkin-Elmer ion trap detector. Vapor phase chromatog-

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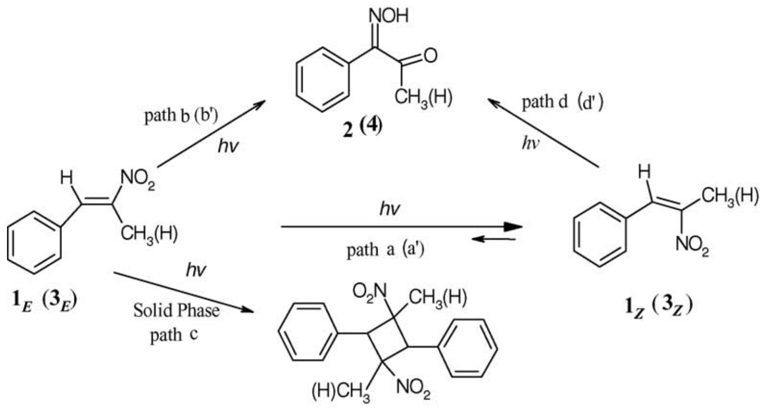


Scheme 1. Photochemistry of geometrical isomers of β-nitrostyrenes: $\mathbf{1}_E$, $\mathbf{1}_Z$, $\mathbf{3}_E$ and $\mathbf{3}_Z$.

raphy is carried out on a Varion model 1720 with disc integrator and temperature programming capability. Melting points are measured using Gallenchamp and are uncorrected.

2.1. Syntheses

Methods of Gaired and Lappin [33] and the Robertson [34] are used for the synthesis of $\mathbf{1}_E$ and $\mathbf{3}_E$.

2.1.1. (E)-β-Methyl-β-nitrostyrene, $\mathbf{1}_E$ [18]

Yellow crystals; mp 63–64 °C (from EtOH); *m/z* 163(M^+ 41.7%), 162(1.8), 146(13.3), 135(10), 117(25.9), 116(45), 115(100), 106(38.3), 91(55), 77(16.7), 63(20). ^{1}H NMR ($CDCl_3$) δ: 2.5(3H, d, Me), 7.2–7.7(5H, m, ArH), 8.1(1H, s); UV λ_{max}(EtOH), 305 nm, ε_{max} 115; yield 60%.

2.1.2. (E)-β-Nitrostyrene, $\mathbf{3}_E$ [32]

Yellow crystals; mp 60–61 °C (from EtOH); *m/z* 148(M^+ 31.7%), 117(23.9), 116(41), 115(100), 106(35.3), 91(45), 77(14.7), 63(20). ^{1}H NMR ($CDCl_3$) δ: 6.8(2H, d), 7.2–7.7(5H, m, ArH), 8.1(2H, d); UV λ_{max}(EtOH), 300 nm, ε_{max} 115; yield 70%.

The authentic sample of $\mathbf{1}_Z$ is synthesized by the procedure of Ono et al. [35]. Treatment of (*E*)-β-methyl-β-nitrostyrene, $\mathbf{1}_E$, with sodium benzeneselenolate, generated in situ from diphenyl diselenide and $NaBH_4$ in ethanol, followed by protonation with acetic acid at −78 °C afforded *erythro*-β-nitroselenide stereoselectively. Treatment of the β-nitroselenide with H_2O_2 at 0 °C resulted in the elimination of benzeneselenic acid to give (*Z*)-β-methyl-β-nitrostyrene, $\mathbf{1}_Z$.

2.1.3. (Z)-β-Methyl-β-nitrostyrene, $\mathbf{1}_Z$ [23,35]

White crystals; mp 50–51 °C (from EtOH). ^{1}H NMR ($CDCl_3$) δ: 2.48(3H, d, Me), 7.2–7.7(5H, m, ArH), 6.6(1H, s); UV λ_{max}(EtOH), 270 nm, ε_{max} 104; yield 20%.

2.2. Photoproducts

A solution is prepared by dissolving 2 g of $\mathbf{1}_E$ (or $\mathbf{1}_Z$) in 550 ml of 95% ethanol. The solution is degassed for one half hour with N_2. The ultraviolet irradiation is stopped after 3 h and the solvent removed under reduced pressure. Photolysis mixture is separated via PTLC; white crystals are obtained.

2.2.1. 1-Phenyl-1,2-propanedione-1-oxime, **2** [18]

White crystals; mp 173 °C (from EtOH); *m/z* 163(M^+ 30%), 158(31), 146(13), 117(23), 116(91), 115(100), 106(33), 91(45), 77(19), 63(17).

^{1}H NMR ($CDCl_3$) δ: 2.5(3H, d, Me), 7.1–7.5(5H, m, ArH); yield 53%.

2.3. Preliminary kinetic studies

Upon irradiation of $\mathbf{3}_E$, both $\mathbf{3}_Z$ and **4** are generated and identified by NMR.

Sample solutions are prepared by dissolving 1.2×10^{-4} mol of $\mathbf{1}_E$, $\mathbf{1}_Z$ or $\mathbf{3}_E$ in 0.5 ml CD_3Cl in quartz NMR tubes and then degassed under argon atmosphere. Irradiation are carried out using similar conditions such as light intensity, with a low-pressure mercury lamp by a monochromatic UV light with $\lambda_{max} = 254$ nm at room temperature. The progresses of the photochemical reactions are monitored by ^{1}H NMR spectroscopy. Changes of nitro-olefins are measured by integration of the corresponding starting materials, intermediates and product peaks.

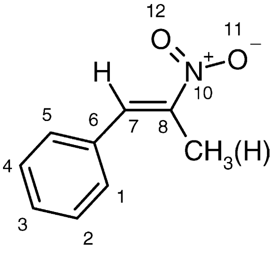
2.3.1. 1-Phenyl-1,2-ethanedione-1-oxime, **4**

^{1}H NMR ($CDCl_3$) δ: 7.2–7.7(5H, m, ArH), 9.7(1H, s).

3. Results and discussion

A serious problem with the previous works on the photochemistry of β-nitrostyrenes is the overwhelming usage of UV analyses, where many details were masked [5–17]. As a follow up on our β-nitrostyrene mechanistic studies [16,17], we have employed a 500 MHz NMR in photolysis of (*E*)-β-methyl-β-nitrostyrene, $\mathbf{1}_E$, (*Z*)-β-methyl-β-nitrostyrene, $\mathbf{1}_Z$, (*E*)-β-nitrostyrene, $\mathbf{3}_E$, and (*E*)-β-nitrostyrene, $\mathbf{3}_Z$ (paths: a, b′, d and d′ in Scheme 1). New

Table 1
Ab initio calculations of torsion angles for $\mathbf{1}_E$, $\mathbf{1}_Z$, $\mathbf{3}_E$, and $\mathbf{3}_Z$



$\mathbf{1}_E$ ($\mathbf{3}_E$)

Torsional angle	Basis set	$\mathbf{1}_E$ singlet	$\mathbf{3}_E$ singlet	$\mathbf{1}_E$ triplet	$\mathbf{3}_E$ triplet	$\mathbf{3}_Z$ singlet	$\mathbf{1}_Z$ singlet	$\mathbf{3}_Z$ triplet	$\mathbf{1}_Z$ triplet
1-6-7-8	Sto-3G	36.73	0.00	46.88	5.09	45.32	43.92	−40.31	34.93
1-6-7-8	6-31G*	44.10	0.00	34.53	33.67	36.01	43.99	47.31	48.61
7-8-10-12	Sto-3G	1.19	0.00	20.06	−21.66	28.13	44.15	−45.81	54.87
7-8-10-12	6-31G*	−5.48	0.00	−0.95	−11.21	7.97	44.21	32.81	41.41
7-8-10-11	Sto-3G	−179.02	180.00	154.35	−155.95	−154.04	−137.81	−176.44	−176.99
7-8-10-11	6-31G*	−174.20	180.00	131.37	−161.13	−173.26	−137.68	−179.34	−172.17

findings in this manuscript reveal intermediacy of $\mathbf{1}_Z$ in photoconversion of $\mathbf{1}_E$ to **2** as well as photo-rearrangements of $\mathbf{1}_Z$ to 1-phenyl-1,2-propanedione-1-oxime, **2**, plus $\mathbf{3}_E$ to 1-phenyl-1,2-ethandione-1-oxime, **4**, at different relative rates. Such details were not recognized before; instead, general statements were made to explain the steric aspects of these reactions. For instance, on the photo-rearrangement of β-methyl-β-nitrostyrene, **1**, to the ketooxime, **2**, it was stated: "steric features of *the molecule* which tend to hold the nitro-group out of the plane of the double bond, i.e., out of conjugation greatly facilitate the reaction" [15]. However, no clear reference to the geometrical isomerization ($\mathbf{1}_E$ versus $\mathbf{1}_Z$) and/or the electronic state of "*the molecule*" (S_0 versus T_1, etc.) was made. One may presume "*the molecule*" they were scrutinizing was $\mathbf{1}_E$ with the electronic state of S_0. It was then suggested that the β-methyl group distorts the planarity of nitro-alkenyl moiety in β-methyl-β-nitrostyrene; while hydrogen was not able to do such a distortion of the planarity to β-nitrostyrene. Consequently, this distortion of planarity, enhances the $n \rightarrow \pi^*$ excitation to the extent where the oximinoketone, **2**, is formed from photo-rearrangement of β-methyl-β-nitrostyrene; while no trace of **4** was detected from the photo-rearrangement of β-nitrostyrene (Scheme 1) [5,15]. However, the X-ray [36] and our semi-empirical calculations did not show any significant difference between the nitro-alkenyl planarity of $\mathbf{1}_E$ compared to $\mathbf{3}_E$. Another word, contrary to what was suggested, β-methyl group in $\mathbf{1}_E$, does not appear to "hold the nitro-group out of the plane of the double bond, i.e., out of conjugation", anymore than the β-hydrogen in $\mathbf{3}_E$. In order to resolve this planarity dilemma,

Irradiation time (t /min)	$\mathbf{1}_E$ %	$\mathbf{1}_Z$ %	**2** %
0	100.0	0.0	0.0
5	87.6	12.4	0.0
10	65.7	34.3	trace
15	45.1	46.9	8.0
20	32.0	53.5	14.5
25	22.9	52.2	24.9
35	18.1	34.7	47.2
40	15.0	24.0	61.0
45	10.1	17.9	72.0
50	0.0	trace	100.0

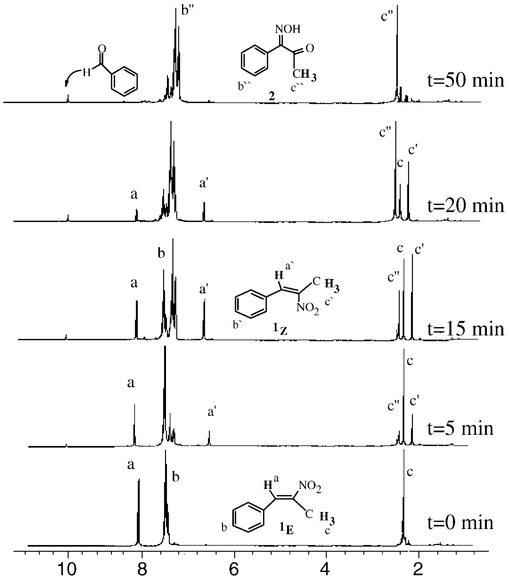


Fig. 1. Tracing ^{1}H NMR spectra of (*E*)-β-methyl-β-nitrostyrene, $\mathbf{1}_E$, photo-isomerization to (*Z*)-β-methyl-β-nitrostyrene, $\mathbf{1}_Z$, followed by photo-rearrangement of $\mathbf{1}_Z$ (and trace $\mathbf{1}_E$) to 1-phenyl-1,2-propanedione-1-oxime, **2**.

Irradiation time (t /min)	1_Z%	2 %	1_E%
0	100.0	0.0	0.0
5	84.0	8.3	7.3
10	79.0	14.5	6.5
15	61.4	38.6	trace
20	43.4	56.6	trace
25	25.1	74.9	trace
35	11.0	89.0	trace
50	trace	100.0	trace

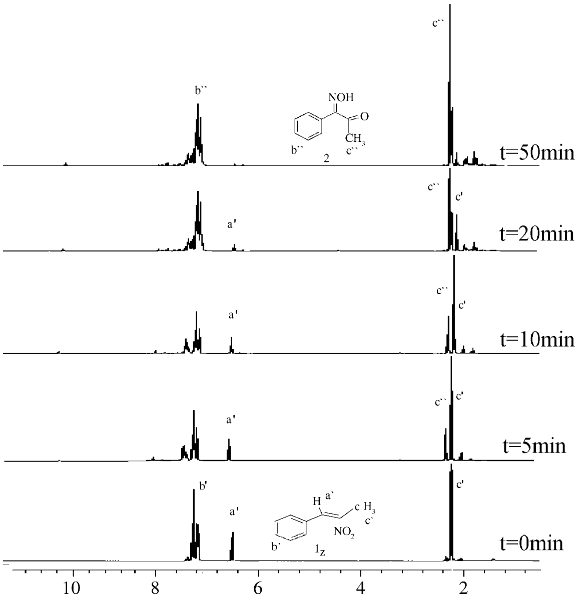


Fig. 2. Tracing ^{1}H NMR spectra of (*Z*)-β-methyl-β-nitrostyrene, $\mathbf{1}_Z$, photo-rearrangement to 1-phenyl-1,2-propanedione-1-oxime, **2**.

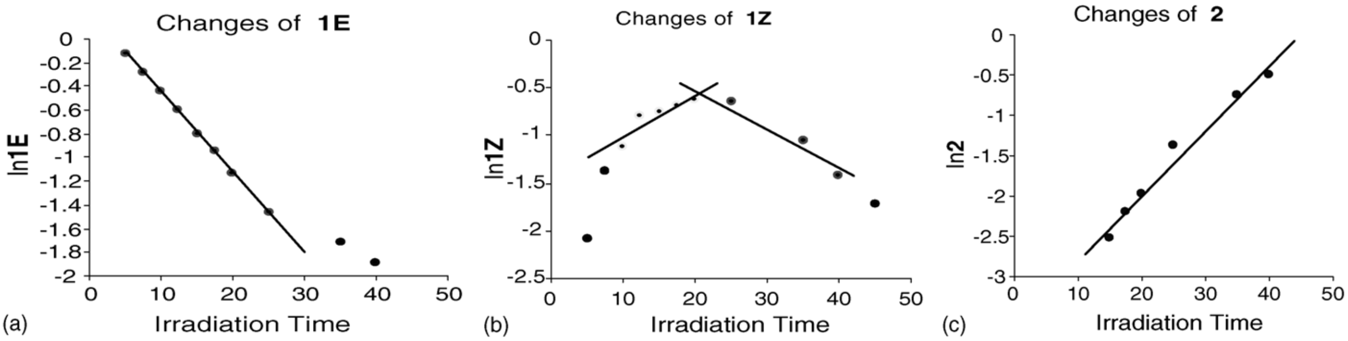


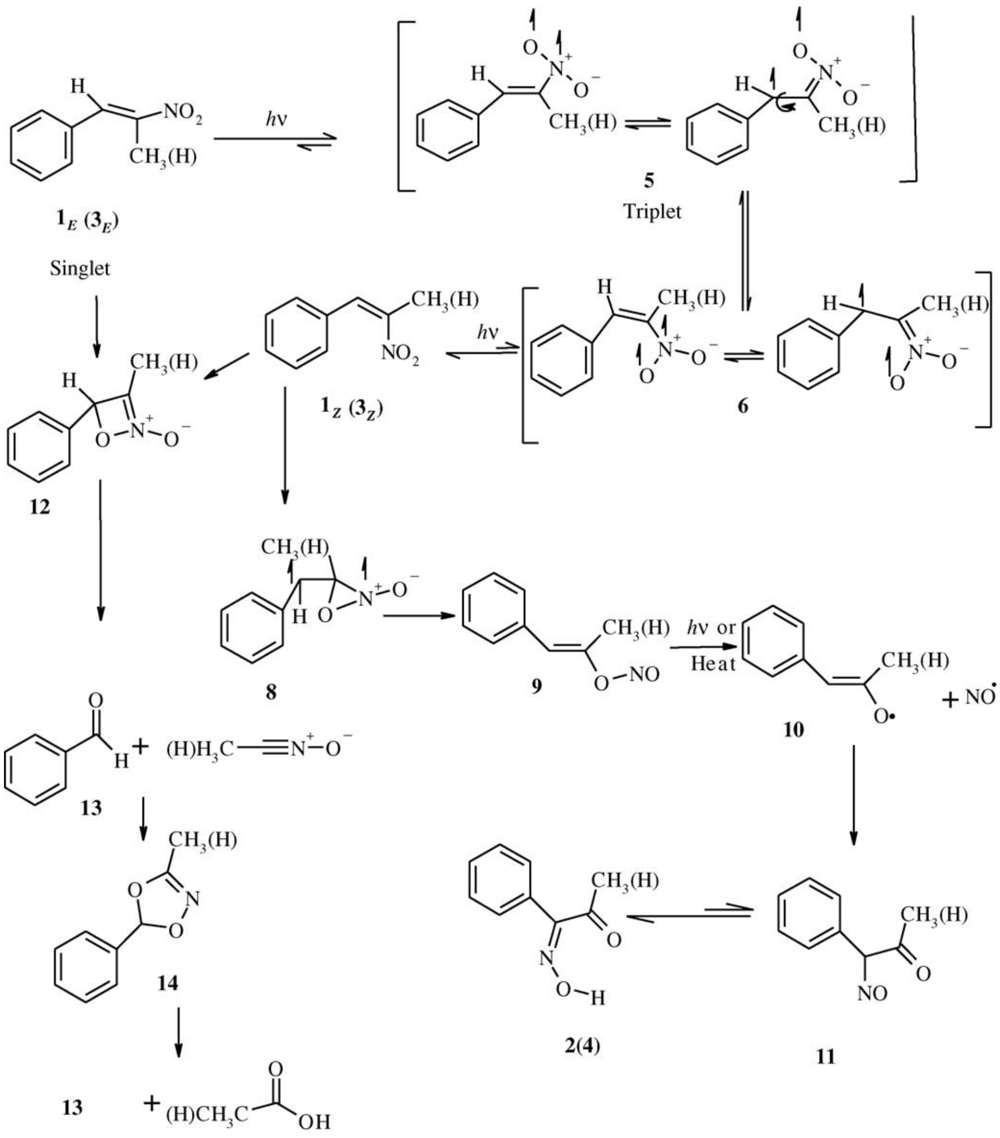
Fig. 3. Changes of $\mathbf{1}_E$, $\mathbf{1}_Z$ and **2** during photolysis of $\mathbf{1}_E$ as a function of irradiation times using similar conditions such as light intensity: (a) Photolysis of pure $\mathbf{1}_E$ as a function of time; (b) photoproduction of $\mathbf{1}_Z$ from $\mathbf{1}_E$ followed by photo-conversion of $\mathbf{1}_Z$ to **2**; (c) photoproduction of **2** from $\mathbf{1}_Z$.

we carried out an ab initio survey at HF/Sto-3g, HF/6-31G* and B3LYP/6-31G* levels of theory optimizing $\mathbf{1}_E$, $\mathbf{3}_E$, $\mathbf{1}_Z$ and $\mathbf{3}_Z$ (Table 1). Amazingly, they do not show much of difference between nitro-alkenyl planarity of ground states (S_0) of $\mathbf{1}_E$ versus $\mathbf{3}_E$. However, geometrical differences are relatively more pronounced for triplets (T_1) of $\mathbf{1}_E$ and $\mathbf{3}_E$ which were not considered before.

In an attempt to solve this problem experimentally, a pure sample of $\mathbf{1}_E$ [24,25] is synthesized and irradiated in ethanol, using a low-pressure mercury lamp with a monochromatic UV light ($\lambda_{max} = 254$ nm). Photochemical changes of $\mathbf{1}_E$ are monitored by a Bruker 500 MHz NMR, every 5 min, through integration of the corresponding ^{1}H NMR peaks. Intensities of $\mathbf{1}_E$ absorption peaks: a, b and c; gradually decrease as a function of time (Fig. 1). Simultaneously, new absorption peaks: a′, b′ and c′ appear which correspond to $\mathbf{1}_Z$ ($t = 20$ min). Finally, absorptions related to **2**: a″, b″ and c″ ($t = 50$ min) come to existence at the expense of disappearing $\mathbf{1}_E$ and $\mathbf{1}_Z$ peaks. Traces of benzaldehyde, **13**, suggested by Matsuura et al. [25], is clearly detected at 9.8 ppm ($t = 40$ min). In order to substantiate photolysis of $\mathbf{1}_E$, an authentic sample of $\mathbf{1}_Z$ is synthesized [35] and irradiated under the same conditions (Fig. 2). Again, the progress of photolysis of $\mathbf{1}_Z$ is monitored via ^{1}H NMR. All $\mathbf{1}_Z$ is converted to **2**, except for its trace isomerization to $\mathbf{1}_E$.

Using similar conditions such as light intensity, ^{1}H NMR tracings preliminary kinetic analysis of conversions $\mathbf{1}_E$ to $\mathbf{1}_Z$ and $\mathbf{1}_Z$ to **2** are carried out (Fig. 3). Reporting ^{1}H NMR (Figs. 1 and 2), instead of ^{13}C NMR spectral results, is to avoid possible integration errors, due to nuclear Overhauser effects (nOe) [37]. Thereby, isomerization of $\mathbf{1}_E$ to $\mathbf{1}_Z$ occurs at a relative rate of $k_{\text{i-Me}} = 0.083\ \text{s}^{-1}$. Both $\mathbf{1}_E$ and $\mathbf{1}_Z$ go through a nitro-nitrite photo-rearrangement to **2** with relative $k_{\text{r-Me}} = 0.011\ \text{s}^{-1}$ and $k'_{\text{r-Me}} = 0.070\ \text{s}^{-1}$, respectively.

A pure sample of $\mathbf{3}_E$ [24,25] is also synthesized and irradiated under similar condition used for $\mathbf{1}_E$. Its ^{1}H NMR peaks, including a doublet at 8.05 ppm (H_{trans}-olefin), gradually decrease while the doublet of newly born $\mathbf{3}_Z$ (H_{cis}-olefin) appears at 6.9 ppm. Meanwhile, the absorptions corresponding to **4** grow. Photo-isomerization of $\mathbf{3}_E$ to $\mathbf{3}_Z$ takes



Scheme 2. Revised provisional mechanism for photochemistry of $\mathbf{1}_E$ and $\mathbf{3}_E$ [18].

place at the relative rate $k_{\text{i-H}} = 0.011\,\text{s}^{-1}$. The relative rates of photo-rearrangements of $\mathbf{3}_E$ and $\mathbf{3}_Z$ to **4** are measured with $k_{\text{r}} = 0.025\,\text{s}^{-1}$ and $k'_{\text{r}} = 0.010\,\text{s}^{-1}$, respectively.

The previously reported possible mechanism shown in Scheme 2 is now further elucidated by our results. This mechanism, while not totally substantiated yet, appears to complete and unify ideas proposed by Chapman [14,17], Pinhey [24], Matsuura [25], Reasoner and us [18,26].

4. Conclusion

Using similar conditions such as light intensity, an unprecedented relative rate is found for *cis–trans* photo-isomerization of (*E*)-β-methyl-β-nitrostyrene, $\mathbf{1}_E$, to (*Z*)-β-methyl-β-nitrostyrene, $\mathbf{1}_Z$ ($k_{\text{i-Me}} = 0.083\,\text{s}^{-1}$). Under the same conditions, relative rates are also found for novel photo-rearrangements of: $\mathbf{1}_Z$ to 1-phenyl-1,2-propanedione-1-oxime, **2** ($k'_{\text{r-Me}} = 0.070\,\text{s}^{-1}$); (*Z*)-β-nitrostyrene, $\mathbf{3}_Z$, to 1-phenyl-1,2-ethandione-1-oxime, **4** ($k'_{\text{r}} = 0.010\,\text{s}^{-1}$); and $\mathbf{3}_E$ to **4** ($k_{\text{r}} = 0.025\,\text{s}^{-1}$). Further ^{1}H NMR preliminary kinetic analysis show a nitro-nitrite photo-rearrangement of $\mathbf{1}_E$ to **2** with relative $k_{\text{r-Me}} = 0.011\,\text{s}^{-1}$. Under the same conditions, photo-isomerization of $\mathbf{3}_E$ to $\mathbf{3}_Z$ takes place at a relative $k_{\text{i-H}} = 0.011\,\text{s}^{-1}$.

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